	Furaca #06	
	AIM: - Preparation of Furaca from 7-ACA by new	
		\$12 55 \$12 55
	reili	
	MODIFICATION:	
	i) 0.15g EDTA added before 7-ACA addition.	F
	2) 0.159 EDTA added later Reaction mases addition	
	3) 25%. Oxcest NaSH.	
	Procedure -	
	Stage I	
2.0	Preparation of TFA	
	RAW MATERIALS '-	
22,24	2 - Furcy chloride : 27.59 (23m).	
	DMW : (350 + 15) ml	
	Nash : 37.59.	
Mary No.	EtnAc : 25c mi	
9	1:14cl : 49 ml. pH - 0.94.	
	DMW : 175 ml.	
e.		
	Nation 209:	
Tion to	Etc Ac : 100 ml	
	EFC AC	
	PROCEDURE	
	1) Time was charged followed by Nash at RT.	
	2) The finnel was washed with DMW.	
	3)2-Furayl Chloride was then added in 40-450 at 20-	
	A) The mexture was estured for 5' and Sample give	
	for monitoring.	
	5) EtoAc was then added and the pt adjusted to 0.9-17	
	with 121 Hil in 15-20	
	6) The lawers were Seperated and OL1 given for HPLC.	
	: #) To one organic taye; DMW was added and the pH	
	adjusted to 7.0-7.2 using Nakeo3 in 15-20' at 20.	
	The mixture stirred for 30' or 20-22's	(8)

	· · · · · · · · · · · · · · · · · · ·
	8) The layers were Separated. To the agreement layer too Ae
	: was added the pH adjusted to 0.9-1.0 using 1:1 HU.
基	9) The layers were separated and one organic layer (0 + 2)
	I taken per next stage after & sample was given por HPLC
	Reaction monitoring
	TFA Imp.
	99.51 -
	DL ₃ 96.50 1.98
	062 96.50 1.98 Observations
	CL1 - 250 mi AL2 - 230 mi CL3 - 130 ml
	Stage II
	Preparation of Funaca.
	RAW MATERIALS
	7- ACA : 50.09.
	EtoAc ; 200 ml.
	G7AA : 30 ml
	BF ₃ : 68.5g
	TFA : 130 ml.
	(
	·
	SH5 : 1.C g
	20% NH3 : 87ml DMW : (50 + 150 + 50) mi, (Spray+shrry+spra
7	Stepper (Special Special Copies & Clurry + Special Spe
1	
	PROCEDURE'-
	: 1) Floric and GAA were Charged and the temperature
	· lawered to Oc.
	2) BF3 was then purged at 15°C.
	3) FDTA was then added.
	4) The mix'ou stored for 5'
7	3 ⁶ , 3,

5) The mirlim Slaves to the completion of the treation of the mirlim Slaves to the completion of the treation was placed followed by the to the treation was then changed followed by the to the treation was then changed followed by the treation was the total followed by the treation was the total followed by the mirror was the treation of the treation of the product was there and washings with the treation of the product was filtered and washings with the treation of the tre			
Example 10 15° Separation Experiments of the treastion; EDTA was added planned by the te Driv Peaction; was then charged followed by SH3: 3) The ph was then adjusted to 3.5 in \$6-45' wains 20° le NH3. At 25-35°C 9) The mixture was settled from \$6' at 25-35°C. 20.25°C 10) The product was filtered and washings with Drive and Ethered given. Tracker monitoring. 7-th Funce TFA Imp. 45' 931 72.77 16.38 0.24. 115' 931 72.77 16.38 0.24. 115' 0.91 38.77. 7.84 0.33. 2 to 15' 0.91 38.77. 7.84 0.33.		5) 7-AcA was then charged followed by IFA and	
EDTA was cooled to 15 c appearance of the treaction. EDTA was added followed by the to the Treaction. Was then charged followed by SHS. S) The ph was then adjusted to 3.5 in 30-45 whing soil NH2 at 25-35 c. 9) The mixture was stoned for 30 at 25-35 c. 20-25 c. 10) The product was filtered and washings with Drive and the stone of the stone		the mixture Swar und as T	
# Before completion of the recetion was then charged followed by SHS: \$) The ph was then adjusted to 3.5 in \$0.45' using so's NH3. at 25-34'. 9) The mixture was stored for \$0' at 25-35's. 20.25's. 10) The product was filtered and washings with DHW. assistings given. **Reaction monitoring.** # PACK Funca TFA Jump. # PACK Funca TFA Ju		1) Day was Cooled to 15 c seperately.	
Followed by SHS. 1		1) After completion of the reaction FDIA was abouted	
Followed by SHS. 1		followed by the to DMW Reaction was then Our you	· · · · · · · · · · · · · · · · · · ·
8) The pt was then adjusted to 3.5 and 42.75 20.16 NH3. at 25.35 c 9) The mixture was subsaid for 30. at 25.25 c. 20-25 c 10) The product was filtered and washings with Thrue and washings with Thrue and washings with Thrue and washings with Thrue and vashings with Th		loughed by SHS.	
9) The mixture was situred for \$8° at \$5.25° (20.25° (8) The pH was then adjusted to 3.5 In 40-45 12mg	
Reaction monitoring. 7-44 Funca TFA Imp. 45' 9.31 72.77 16.38 0.24 . 1 125' 1.75 87.2 8.67 0.33. 2 12 15' 0.91 88.77 7.84 0.23 West wit: 116.8 9. This Furace is used for court 0.5	1		
Reaction monitoring. 7-44 Funca TFA Imp. 45' 9.31 72.77 16.38 0.24 . 1 125' 1.75 87.2 8.67 0.33. 2 12 15' 0.91 88.77 7.84 0.23 West wit: 116.8 9. This Furace is used for court 0.5		9) The mixture was storred for so at the with DMW	
Reaction monitoring. 7-ACA Furaca TEA Jomp. 45' 9.31 72.77 16.38 0.24 . 1hr. 45' 1.75 57.2 8.67 0.33. 2hr. 15' 0.91 88.77 7.84 0.23 week we : 116.89. This Furaca is used for Crur \$ 05		10) The product was filtered and washings	
7-ACA Funaco TFA Jump. 45' 9.31 72.77 16.38 0.24 1 hr 45' 1.75 37.2 8.67 0.33 2 hr 15' 0.91 38.77 7.84 0.33 West we : 116.8 9 This Fur Area is used for Crur \$ 0.5		ancestitute given.	
7-ACA Funaco TEA Jomp. 45' 9.31 72.77 16.38 0.24 1 hr 45' 1.75 37.2 8.67 0.33 2 hr 15' 0.91 88.77 7.84 0.33 West We : 116.89 This Fur Area is used for Crur \$ 0.5			
1.5' 9.31 72.77 16.38 0.24 . 1.125 37.2 8.67 0.33 2.125' 0.91 88.77 7.84 0.33			
1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1		7-464	
1 hr 45' 1.75 54.20 1.84 0.33 2 hr 15' 0.91 88.77. 7.84 0.33 0.33 0.91 0.45 0.4		45 9.31 7-1.	
This Furner is used for Cfur \$ 05		1 hr 45' 1.75 57 2 3.31 0.33	
This Furnea is used for Crur \$ 05		2 hr 15' 0.91 88.77 7 84	
This Furnea is used for Crur \$ 05			
This Furnea is used for Crur \$ 05			
		wet we . 116.8 g	
	3	- Crur # 05	
		This Former is worth	
	3.22		
			<u> </u>
			1
			Age
			1
	policia pa		